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FURTHER PRECIPITATION REACTIONS ASSOCIATED WITH  
 $\beta'$  ( $\text{Al}_3\text{Zr}$ ) PARTICLES IN  $\text{Al-Li-Cu-Mg-Zr}$  ALLOYS

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 $\beta'$  ( $\text{Al}_3\text{Zr}$ ) PARTICLES IN Al-Li-Cu-Mg-Zr ALLOYS

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C. N. J. Tite\*

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SUMMARY

Transmission electron microscopy has been used to investigate the precipitation reactions associated with  $\beta'$  ( $\text{Al}_3\text{Zr}$ ) particles in 8091 alloy sheet (<2 mm thick). The microstructures obtained at various stages of ageing have been studied. It was observed that in addition to acting as nucleation sites for the precipitation of  $\delta'$  ( $\text{Al}_3\text{Li}$ ), the  $\beta'$  particles also provide sites for the nucleation of dislocation loops. These loops then act as sites for the heterogeneous precipitation of S phase ( $\text{Al}_2\text{CuMg}$ ) during ageing. An additional type of precipitate at the  $\beta'$  sites has been identified as  $\theta$  ( $\text{Al}_2\text{Cu}$ ).

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## LIST OF CONTENTS

	Page
1 INTRODUCTION	3
2 EXPERIMENTAL PROCEDURE	3
3 RESULTS	3
4 DISCUSSION	5
5 CONCLUSIONS	5
References	6
Illustrations	Figures 1-6
Report documentation page	inside back cover

**A-1**



## 1 INTRODUCTION

In 8090 and 8091 Al-Li-Cu-Mg-Zr alloys, as in other high-strength aluminium alloys (eg 7010, 7050), zirconium is added to provide grain refinement and to inhibit recrystallisation in wrought products. This behaviour is reported to be associated with the presence of a fine (20-30 nm) but non-uniform distribution of coherent  $\text{Al}_3\text{Zr}$  ( $\beta'$ ) particles in a metastable cubic ( $\text{Ll}_2$ ) form; these particles form during solidification and remain stable during subsequent thermal and mechanical treatments<sup>1,2</sup>. The role of  $\beta'$  particles in Al-Li alloys is essentially similar<sup>3</sup>, but the particles play an additional role in providing heterogeneous nucleation sites for the precipitation of  $\delta'$  ( $\text{Al}_3\text{Li}$ ) during age hardening of Al-Li based alloys<sup>4,5</sup>. Furthermore  $\beta'$  particles have been observed to act as preferential sites for the precipitation of  $\theta$  ( $\text{Al}_2\text{Cu}$ ) and  $\text{T}_1$  ( $\text{Al}_2\text{CuLi}$ ) in an Al-Li-Cu alloy with a high copper to lithium ratio<sup>6</sup>. The present paper reports observations of further microstructural changes associated with  $\beta'$  particles during age hardening of Al-Li-Cu-Mg-Zr alloys conforming to the 8091 specification.

## 2 EXPERIMENTAL PROCEDURE

Alloy sheet (<2 mm thick) conforming to the 8091 specification (Al-2.6 Li-1.9Cu-0.8Mg-0.12Zr nominal composition) was solution treated at 540°C and cold water quenched. Microstructural examination was undertaken at various stages in the subsequent ageing treatments. Thin foils for Transmission Electron Microscopy (TEM) were prepared via electrolytic thinning in 30%  $\text{HNO}_3$  in methanol. TEM was undertaken on a JEOL 200 CX operating at 200 kV. High spatial resolution (3 nm) X-ray microanalysis was undertaken on a Philips 420T TEM operating in nanoprobe mode with an accelerating voltage of 135 kV.

## 3 RESULTS

In the as-quenched condition the microstructure consisted of a non-uniform distribution of  $\beta'$  particles, together with a widespread and uniform precipitation of very fine  $\delta'$  particles (<5 nm). The number of quenched-in defects such as dislocation loops and helices was low compared with lithium-free alloys; these observations have previously been attributed to the high-lithium atom to vacancy binding energy combined with the high concentration of lithium atoms<sup>7</sup>.

During natural ageing the number of dislocation loops and helices was found to increase; this is consistent with previous results<sup>8</sup>. The dislocation loops, however, were often associated with the  $\beta'$  particles as shown in Fig 1. Analysis of the loops using different reflecting vectors indicated that loops have a

Burgers vector  $a/2 \langle 110 \rangle$  (ie there is no dislocation contrast for  $\bar{g} = \bar{1}11$ ) - Fig 1; the dislocation loops are therefore glissile. Furthermore, the dark field image of the same specimen area using  $\delta'$  superlattice reflection reveals an enhanced precipitation of very fine  $\delta'$  at some of the  $\beta'$  particles; subsequent ageing results in the coalescence of these fine particles to form the complete  $\delta'$  coating of the  $\beta'$  particles which has been previously observed.

During heating up to the ageing temperature the dislocation loops surrounding the  $\beta'$  particles grow, and there is also evidence of multiple loop formation at some of these sources - Fig 2. During age-hardening these enlarged loops act as nucleation sites for the heterogeneous precipitation of S phase ( $Al_2CuMg$ ) - Fig 3; precipitation at such sites in Al-Cu-Mg alloys is well documented<sup>9</sup>.

Besides the loop formation and subsequent heterogeneous precipitation of S-phase, there is evidence of an additional reaction at the  $\beta'$  sites during artificial ageing, giving rise to particles which are revealed by inelastic scattering contrast - Fig 4. These additional particles were similarly orientated to the dislocation loops at the  $\beta'$  sites, and often the loops were visible around the particles. X-ray microanalysis of these particles together, with analyses from the associated  $\beta'$  particle and the adjacent matrix are summarised in Fig 5. There is a higher copper concentration within the particles than in the surrounding matrix.

Diffraction evidence from the thin copper rich discs was difficult to obtain. However, 'd' spacings from  $\langle 001 \rangle$ ,  $\langle 011 \rangle$ ,  $\langle 111 \rangle$  and  $\langle 112 \rangle$  diffraction patterns of an area containing a high density of these particles were measured - Fig 6: the values corresponding to precipitate reflections are recorded as a frequency histogram in Fig 6a where the numbers of diffraction spots consistent with a particular interplanar spacing are normalised for the complete selection of reflections encountered from the selected area of the sample. By subtracting the calculated normalised frequencies of 'd' spacings which correspond to phases which are known to be present, namely  $\delta'$ , S, and  $T_1$ , the distribution for the 'd' spacings remaining is shown in Fig 6b; these presumably correspond to the copper rich phase present in the area. Comparison of the frequency distribution of Fig 6b with calculated distributions for  $Al_2MgLi$ ,  $T_B(Al_7Cu_4Li)$ ,  $\theta'(Al_2Cu)$  and  $\theta(Al_2Cu)$  reveal that the distribution for  $\theta$  matched most closely with the diffraction evidence - Fig 6c.

#### 4 DISCUSSION

The formation of dislocation loops at  $\beta'$  sites can be explained in terms of a higher vacancy concentration in the vicinity of these particles. The preferential precipitation of  $\delta'$  at this position will lead to an increased concentration of free vacancies which can subsequently condense to form the dislocation loop. These loops have been shown to be glissile with a Burgers vector of the type  $a/2 \langle 110 \rangle$  and may therefore, be able to expand in their own plane as ageing progresses, or else expand by a climb mechanism as further vacancies are released.

Precipitation of S phase is known to occur preferentially at dislocation sites within the Al-Li-Cu-Mg-Zr alloys<sup>10</sup>; the present formation of S precipitates at these dislocation loops is a further example. In the present work this effect has been exaggerated by the use of a slow heating rate to the ageing temperature; under such conditions the loops can expand before significant precipitation of S phase occurs. The development of a balanced combination of properties in 8090 and 8091 relies on the coprecipitation of  $\delta'$  and S phases. Under circumstances in which the use of cold work to promote S phase precipitation is not feasible, the distribution of  $\beta'$  particles may have a significant influence on S precipitates and therefore, on the properties of the age-hardened material.

The present observations of  $\theta$  formation at the  $\beta'$  particles may also influence S precipitation. The homogeneous precipitation of S phase within 8091 has been shown to be critically dependent upon copper and magnesium supersaturation<sup>11</sup>; formation of copper rich  $\theta$  at the  $\beta'$  sites will thus reduce this supersaturation and suppress S formation.

The precise mechanism for  $\theta$  formation at the  $\beta'$  particles is still unclear but the observations of similar loop and particle orientation together with the contrast effects of the particles suggest that there is an infiltration of copper atoms into the dislocation loops, and that this copper rich zone subsequently transforms to  $\theta$ .

#### 5 CONCLUSIONS

The  $\beta'$  particles within Al-Li-Cu-Mg-Zr alloys have been found to provide sites for the formation of dislocation loops which act as sites for heterogeneous S precipitation during age-hardening. Additional precipitation at the  $\beta'$  sites has been identified as  $\theta(\text{Al}_2\text{Cu})$ .

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Fig 1

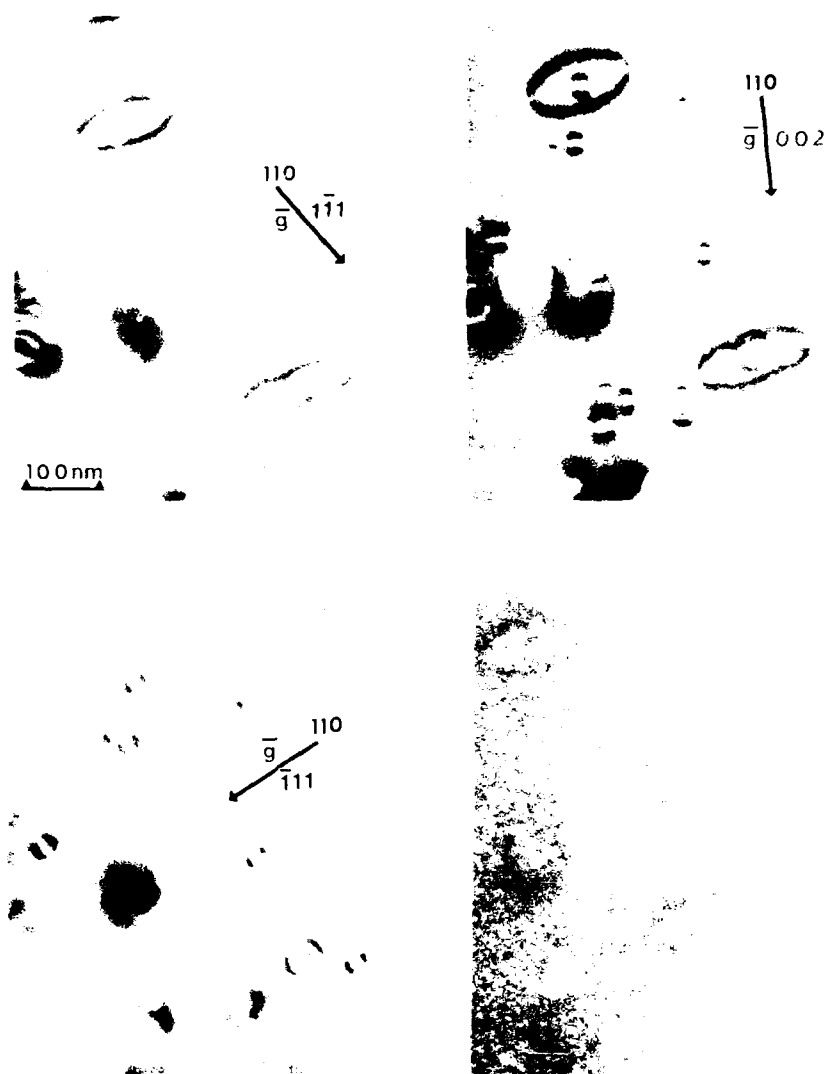


Fig 1 Dislocation loops around  $\beta'$  particles (outlined by strain fields) in as quenched material. The same field of view is shown using three reflecting vectors, and also in dark field using a  $\bar{g}$  diffraction spot.

Figs 2-4

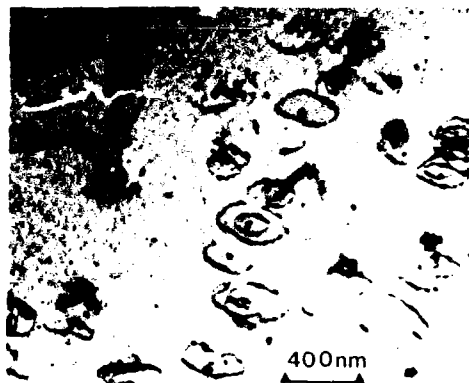


Fig 2 Dislocation loops after heating up to 210°C at 10°C/min;  $\langle 110 \rangle$  Al orientation

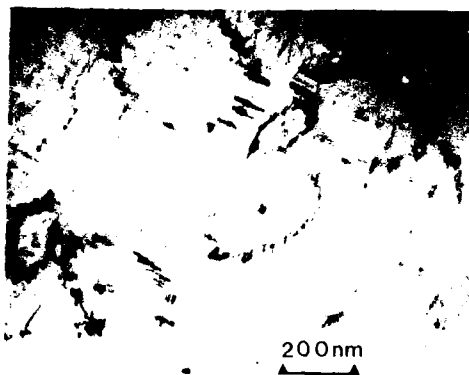


Fig 3 Heterogeneous S precipitation on dislocation loops;  $\langle 110 \rangle$  Al orientation

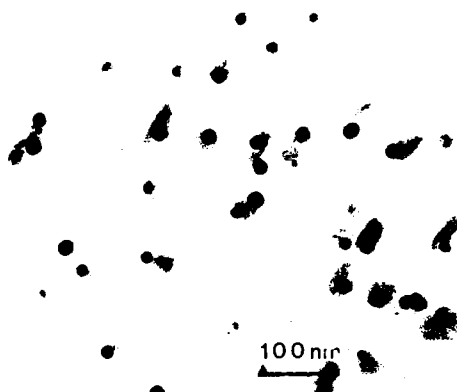


Fig 4 Additional particles at B' sites;  $\langle 110 \rangle$  Al orientation

Fig 5a-c

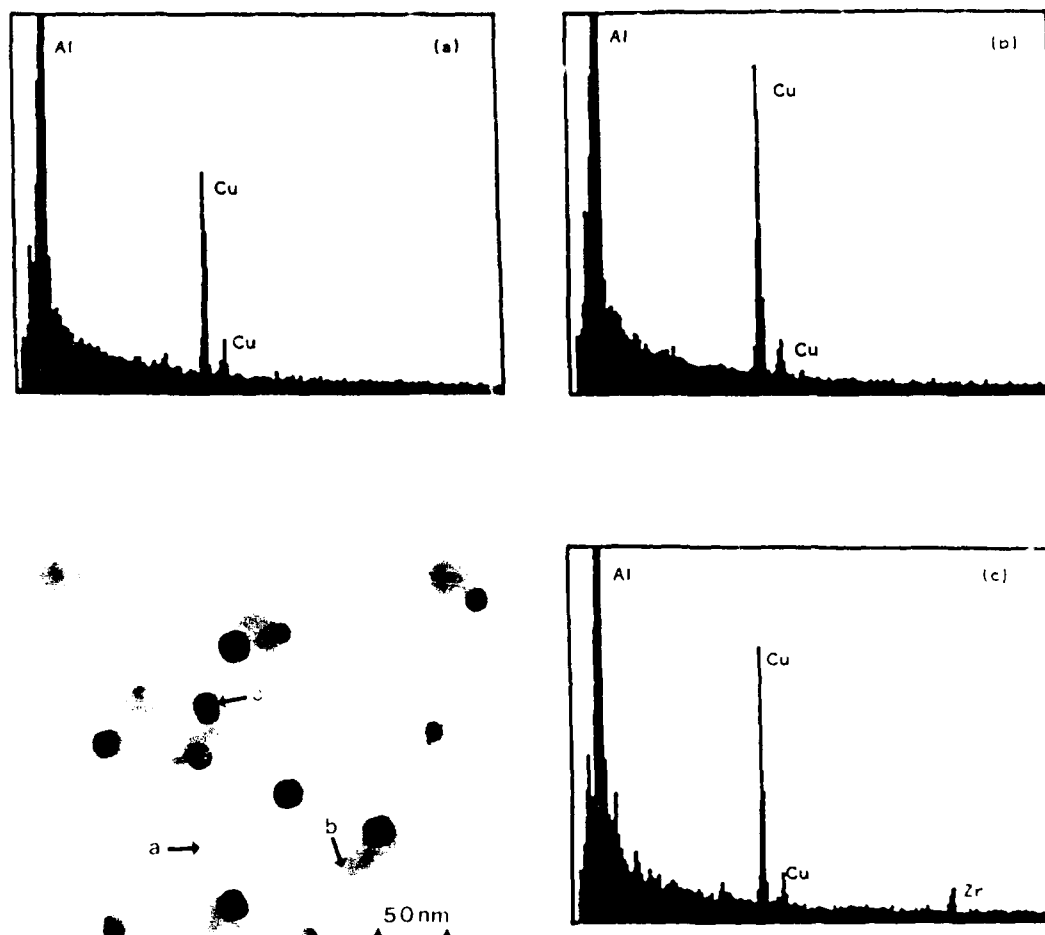


Fig 5 X-ray microanalysis from (a) matrix,  
(b) additional particle and  
(c) R' particle

Fig ba-c

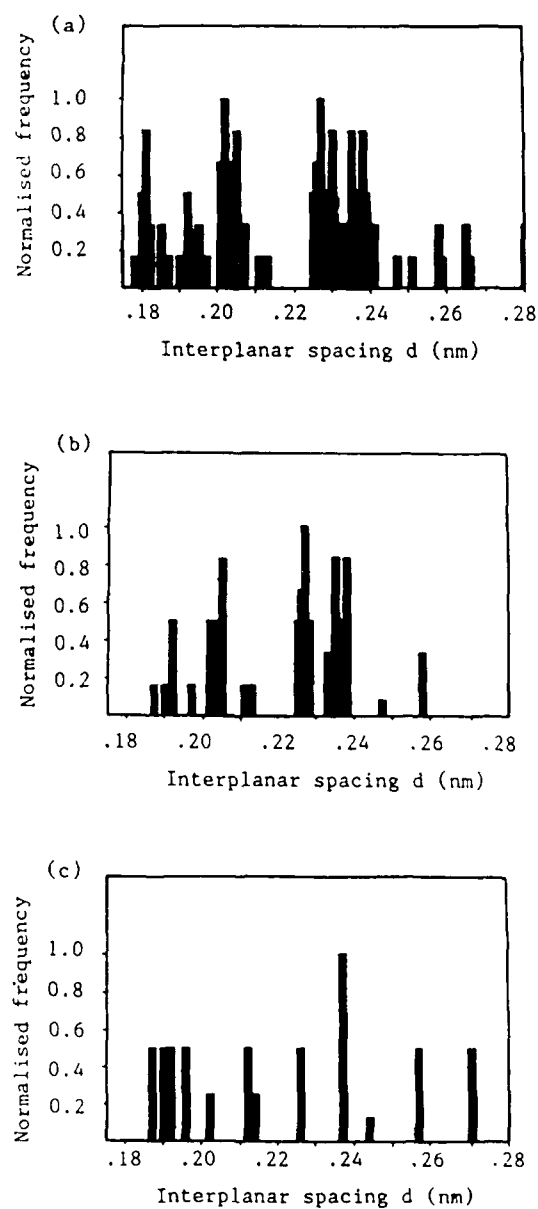


FIG. 6. Frequency histogram of interplanar spacings from electron diffraction patterns: (a) as recorded from a region containing additional particles, (b) the same histogram stripped of  $\delta'$ , S and  $T_1$  precipitate reflections and (c) the theoretical histogram for  $\theta$ .

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